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IS 13651 (1993): 1-1-1 Trichloroethane (methyl chloroform) for electroplating [CHD 5: Electroplating Chemicals and Photographic Materials]

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विद्युत लेपन के लिए 1-1-1 ट्राइक्लोरोइथेन (मिथाइल क्लोरोफार्म) — विशिष्टि

Indian Standard

1-1-1 TRICHLOROETHANE (METHYL CHLOROFORM) FOR ELECTROPLATING — SPECIFICATION

UDC 661·723·63 : 621·357·7

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

May 1993

Price Group 3

Electroplating Chemicals Sectional Committee, CHD 05

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Electroplating Chemicals Sectional Committee had been approved by the Chemical Division Council.

1-1-1 Trichloroethane (Methyl Chloroform) is among the least hazardous of chlorinated solvents widely used as an industrial solvent particularly for metal degreasing operations. It has an excellent solvent power, is non-inflammable and least toxic compared to the other solvents which are being used today. The operating temperature in vapour degreasing process is much lower and thus results in reduction of solvent loss, consumption and cost. It also dissolves readily the common industrial soils without attacking a majority of plastics, resins, sealants and other sensitive materials. This optimum solvency has a great advantage in cleaning/degreasing of electronic and electrical components like relays, resistors, capacitors, transformers, motors, circuit breakers, armature shafts, etc, hermetic stators, precision bearings and other metal parts of precision instruments.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

1-1-1 TRICHLOROETHANE (METHYL CHLOROFORM) FOR ELECTROPLATING — SPECIFICATION

1 SCOPE

This standard prescribes requirements, methods of sampling and tests for inhibited (stabilized) 1-1-1 Trichloroethane (Methyl Chloroform) suitable for degreasing process, and as solvent for oils, fats, greases, waxes, bitumen, tar, gums, resins, etc.

2 REFERENCES

The following Indian Standards are the necessary adjuncts to this standard:

IS No.	*Title*
170 : 1976	Acetone (*second revision*)
245 : 1970	Trichloroethylene, technical (*second revision*)
265 : 1976	Hydrochloric acid (*second revision*)
737 : 1974	Wrought aluminium and aluminium alloys (*second revision*)
1070 : 1992	Reagent grade water (*third revision*)
2362 : 1973	Determination of water by the Karl Fischer method (*first revision*)
5298 : 1983	Method for determination of distillation range and distillation yield (*first revision*)

3 REQUIREMENTS

3.1 Description

3.1.1 1-1-1 trichloroethane shall be colourless liquid, insoluble in water, soluble in alcohol and ether, free from undissolved water and matter in suspension, and shall conform essentially to the formula, CH_3CCl_3.

3.1.2 The inhibitors and impurities shall not materially increase the toxicity of the uninhibited material.

3.2 Scratch Test

The material when tested in accordance with the method given in Annex A shall pass the test.

3.3 The material shall also comply with the requirements prescribed in Table 1.

Table 1 Requirements for 1-1-1 Trichlorethane for Electroplating

Sl No.	Characteristic	Requirement	Method of Test Ref to	
			Annex	Clause No. of IS
(1)	(2)	(3)	(4)	(5)
i)	Purity, percent, *Min*	90	—	8.2 of IS 5298 : 1983
ii)	Colour, Hazen Units, *Max*	15	B	—
iii)	Relative density at 27/27°C	1.297 to 1.319	C	—
iv)	Distillation yield, percent by volume (*see* Note)		—	8.2 of IS 5298 : 1983
	Up to 71 °C, *Max*	5		
	Up to 84 °C, *Min*	95		
v)	Residue on evaporation, ppm, *Max*	10	D	—
vi)	Water, ppm, *Max*	150	—	IS 2362 : 1973
vii)	Acidity (as HCl), ppm, *Max*	10	E	—
viii)	Acid acceptance value (as NaOH), percent by mass, *Min*	0.1	F	—
ix)	Corrosive action on Aluminium	Shall not show any sign of corrosion	G	—
x)	Stability test under reflux percent by mass of hydrochloric	Shall not show any acidity exceeding the equivalent of 0.1	—	A-7 of IS 245 : 1970

NOTE — The correction factor for temperature for a pressure of 760 mm Hg is as follows :

a) For every 10 mm above 760 mm Hg, add 0.43°C to the specified temperature, and

b) For every 10 mm below 760 mm Hg, subtract 0.43 °C from the specified temperature.

4 PACKING AND MARKING

4.1 Packing

The material shall be supplied in sealed and air-tight mild steel or coated galvanized steel drums or high modulus, high density polyethylene drums. The contents may be from 25 kg to 250 kg per container. The material shall be protected from light and stored in cool, dark and well ventilated place.

4.2 Marking

The containers shall be marked with the following particulars:

a) Name of the material;
b) Net mass;
c) Indication of the source of manufacture;
d) Batch number; and
e) Date of manufacture.

5 SAMPLING

5.1 Representative samples of the material shall be drawn and their conformity to this specification shall be determined as prescribed in Annex H.

6 TEST METHODS

6.1 Tests shall be conducted as prescribed in **3.2** and col 4 and 5 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1992) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

(*Clause* 3.2)

METHOD OF TEST FOR SCRATCH TEST

A-1 OUTLINE OF THE METHOD

A-1.1 This method shows if sufficient stabilizer is present.

A-2 PROCEDURE

A-2.1 Place a strip or coupon of aluminium in a sample of used or recovered trichloroethane solvent. Scratch a submerged area of the aluminium with a sharp object using heavy pressure. Don't expose scratches to air during test.

A-3 OBSERVATION

A-3.1 If the scratches don't turn red, enough stabilizer is present. If there is any bleeding, indicated by dark red colour, leave the aluminium in the solvent and see if the scratches heal. If they don't heal, enough stabilizer is not present.

ANNEX B

[*Table* 1, *Sl No.* (ii)]

METHOD OF TEST FOR COLOUR

B-1 APPARATUS

B-1.1 Two matched Nessler cylinders of 100 ml capacity each.

B-1.2 One-mark volumetric flasks of 250 ml and 500 ml capacity.

B-2 REAGENTS

B-2.1 Cobaltous Chloride Hexahydrate

B-2.2 Hydrochloric Acid, Concentrated (*see* IS 265 : 1976).

B-2.3 Chloroplatinic Acid Reagent

Dissolve 250 mg of platinum in a small quantity of aqua-regia contained in a glass or porcelain basin by heating on a water bath. When the metal has dissolved completely, evaporate the solution to dryness. Add 1 ml of hydrochloric acid and again evaporate to dryness. Repeat this operation twice.

B-3 PROCEDURE

B-3.1 Preparation of Colour Standard

Dissolve 0.5 g of the cobaltous chloride hexahydrate and the whole of the chloroplatinic acid (*see* **B-2.3**) in 50 ml of the hydrochloric acid. Warm, if necessary to obtain a clear solution and after cooling, pour into the 500 ml volumetric flask. Dilute with water to the mark. Pipette 7.5 ml of this solution into the 250 ml volumetric flask. Dilute with water to the mark. This diluted solution has a colour of 15 Hazen units and shall always be prepared fresh.

B-3.2 Fill one of the nessler cylinders to the mark with the sample, and the other with the colour standard. Using a white background compare the colours. The colour produced by the sample shall not be darker than the standard colour solution.

ANNEX C

[*Table* 1, *Sl No.* (iii)]

METHOD OF TEST FOR DETERMINATION OF RELATIVE DENSITY

C-1 APPARATUS

C-1.1 Relative Density Bottle

25 ml capacity.

C-1.2 Water-Bath

Capable of being maintained at 27.0 ± 0.2° C.

C-1.3 Thermometer

Any convenient thermometer of a suitable range with 0.1° sub-division.

C-2 PROCEDURE

C-2.1 Clean and dry the relative density bottle, weigh and then fill with freshly boiled and cooled water at 25°C. Fill to overflow in such a manner as to prevent entrapment of air bubbles. Insert the stopper and immerse the bottle in the water- bath. Keep the entire bulb completely covered with water and hold at that temperature for 30 minutes. Carefully remove any water which has exuded from the capillary opening. Remove from the bath, wipe completely, cool and dry the relative density bottle. Using the material under test, proceed exactly as in the case of water and weigh the bottle with the material.

NOTE — Relative density could be determined at the ambient temperature (it is not necessary to maintain the temperature at 27°C) and then converted at 27/27°C by applying a correction factor of + 0.001 1 for each degree celcius fall in temperature and −0.001 1 for each degree rise in temperature.

C-3 CALCULATION

$$\text{Relative density at } 27/27^\circ\text{C} = \frac{A - B}{C - B}$$

where

A = Mass in g of the relative density bottle filled with the material.

B = Mass in g of the clean and dry relative density bottle, and

C = Mass in g of the relative density bottle filled with water.

ANNEX D

[*Table* 1, *Sl No.* (v)]

METHOD OF TEST FOR DETERMINATION OF RESIDUE ON EVAPORATION

D-1 APPARATUS

D-1.1 Evaporating Basin — Made of platinum, silica or borosilicate glass.

D-1.2 Water Bath

D-1.3 Oven —Capable of being maintained at 105 ±2°C.

D-2 PROCEDURE

D-2.1 Evaporate, in the weighed evaporating basin and in small portions at a time, 100 g of the sample of dryness on a boiling water bath containing water. Dry the residue for 1 hour in an oven at a temperature of 105 ± 2°C. Cool in a desiccator and weigh.

D-3 CALCULATION

Residue on evaporation, parts per million

$$= \frac{M_1}{M_2} \times 10^6$$

where

M_1 = mass, in g of residue, and

M_2 = mass, in g of sample taken for test.

ANNEX E

[*Table* 1, *Sl No.* (vii)]

METHOD OF TEST FOR DETERMINATION OF ACIDITY

E-1 REAGENTS

E-1.1 Sodium Hydroxide

0.01 N solution, freshly prepared.

E-1.2 Bromophenol Blue Indicator

Dissolve 0.1 g of the solid material in a mixture of 80 ml of distilled water and 20 ml of denatured alcohol and mix well.

E-1.3 Neutralized Water

Measure 100 ml of water into a flask. Add a few drops of the bromophenol blue indicator and, if necessary, neutralize by dropwise addition of the sodium hydroxide solution or dilute hydrochloric acid solution.

E-2 PROCEDURE

E-2.1 Place 100 ml of the neutralized water in a 250 ml glass stoppered conical flask. Add 100 ml of the sample and shake vigorously. Allow the layers to separate, add 0.5 ml of the bromophenol blue indicator and titrate with the 0.01 N sodium hydroxide solution using a microburette.

E-3 CALCULATION

Acidity, as hydrochloric acid (HCl), parts per million

$$= \frac{3.65 \times T_1}{d}$$

where

T_1 = volume in ml, of 0.01 N sodium hydroxide used, and

d = relative density of the sample.

ANNEX F

[*Table* 1, *Sl No.* (viii)]

METHOD OF TEST FOR DETERMINATION OF ACID ACCEPTANCE VALUE

F-1 OUTLINE OF METHOD

F-1.1 The sample is treated with a mixture of dioxan and hydrochloric acid, and the excess acid is titrated with a standard methanolic sodium hydroxide solution using cresol red as indicator.

F-2 REAGENTS

F-2.1 Sodium Hydroxide —0.1 N methanolic solution.

F-2.2 Dioxan-Hydrochloric Acid Mixture

Add 0.8 ml of concentrated hydrochloric acid to 100 ml of dioxan. Shake well in a glass stoppered flask until the mixture is clear. (This mixture shall be prepared fresh for each test).

F-2.3 Cresol Red Indicator Solution

0.2 g/l.

F-2.4 Neutralized Ethanol

Titrate ethanol with the 0.1 N methanolic sodium hydroxide solution, using cresol red indicator, until the colour of the solution just turns violet.

F-3 PROCEDURE

F-3.1 Transfer, by means of a pipette, 25 ml of the dioxanhydrochloric acid mixture to a 250 ml conical flask having a ground glass neck, and add 10 ml of the sample. Stopper the flask, swirl to mix the contents and leave to stand for 15 minutes at room temperature. Add 25 ml of the neutralized ethanol and titrate the unreacted hydrochloric acid with the 0.1 N methanolic sodium hydroxide solution until a violet colour is obtained and persists for 30 seconds. (Ignore the colour change of the cresol red indicator from pink to yellow). Carry out a blank test on the reagents, omitting only the sample.

F-4 CALCULATION

Acid acceptance value, as sodium hydroxide, (NaOH),

$$\text{percent by mass} = \frac{0.04 \times (V_1 - V_2)}{d}$$

where

V_1 = volume in millilitres, of 0.1 N sodium hydroxide used in the blank test;

V_2 = volume, in millilitres, of 0.01 N sodium hydroxide used for the titration of the sample; and

d = relative density of the sample.

ANNEX G

[*Table* 1, *Sl No.* (ix)]

METHOD OF TEST FOR CORROSIVE ACTION OF ALUMINIUM

G-1 MATERIALS

G-1.1 Aluminium Alloy Sheet HS 15 (*See* IS 737 : 1974).

G-1.2 Fine Emery Cloth

120 grade.

G-1.3 Acetone— (*see* IS 170 : 1976).

G-2 PROCEDURE

G-2.1 Cut a panel, 75 mm x 12.5 mm from the aluminium alloy sheet and polish it with fine emery cloth until it is uniformly bright and almost free from oxide film; thereafter do not touch the panel with the bare hands until completion of the test. Wash the panel with soap and water; rinse in distilled water, then in acetone, and allow to dry. Place the panel in a 150 ml conical flask with a ground glass neck; add 50 ml of the sample, attach a reflux condenser for 5 h. Allow to cool and examine the panel for signs of corrosion.

ANNEX H

(*Clause* 5.1)

SAMPLING OF 1-1-1 TRICHLOROETHANE FOR ELECTROPLATING

H-1 GENERAL REQUIREMENTS FOR SAMPLING

H-1.1 The sampling instruments shall be clean and dry.

H-1.2 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

H-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by shaking or stirring or both by suitable means or by rolling.

H-1.4 The samples shall be placed in suitable, clean, dry and airtight metal containers, or dark or amber glass containers on which the material has no action.

H-1.5 The sample containers shall be of such a size that they are almost completely filled by the sample.

H-1.6 Each sample container shall be sealed airtight after filling and marked with full details of sampling, and lot or batch number.

H-1.7 Samples shall be stored in the cool and dark.

H-2 SAMPLING INSTRUMENT

H-2.1 The following forms of sampling instrument may be used:

a) Sampling bottle or can for taking samples from tanks or drums, and

b) Sampling tube for taking samples from bottles or small containers.

H-2.1.1 *Sampling Bottle or Can*

It consists of a weighed glass or metal container with removable stopper or top to which is attached a light chain (*see* Fig.1). The bottle or can is fastened to a suitable pole. For taking a sample, the bottle or can is lowered into the tank to the required depth and the stopper is then removed by means of the chain.

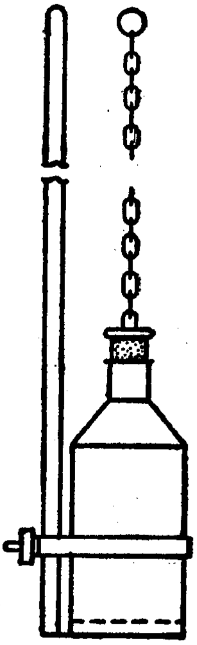

FIG. 1 SAMPLING BOTTLE OR CAN

H-2.1.2 *Sampling Tube*

Made of metal or thick glass, 20 to 40 mm in diameter and 400 to 800 mm in length (*see* Fig. 2). The upper and lower ends are conical and reach 5 to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end.

H-2.1.2.1 For small containers the size of the sampling tube may be altered suitably.

H-3 SCALE OF SAMPLING

H-3.1 For Tanks and Large Drums

Each tank or drum shall be sampled separately.

H-3.2 For Bottles and Small Containers

Each lot (*see* **H-3.2.1**) shall be sampled separately.

H-3.2.1 *Lot*

In any consignment of one type of the material, all the containers of the same size and drawn from a single batch of manufacture shall constitute a lot. If a consignment of one type of the material is known to consist of different batches of manufacture or different sizes of containers, the containers belonging to the same batch and size shall be grouped together and each such group shall constitute a separate lot.

H-3.2.2 The number of containers (n) to be selected from a lot shall depend upon the size of the lot (N) and shall be in accordance with col 1 and 2 of Table 2.

Table 2 Number of Containers to be Selected from Lot of Different Sizes

Lot Size	No. of Containers to be Selected
(1)	(2)
N	n
Up to 15	3
16 to 40	4
41 to 65	5
66 to 110	7
111 and above	10

H-3.2.3 The containers shall be selected at random from the lot and in order to ensure randomness of selection, the procedure given in IS 4905 : 1968 'Methods for random sampling' may be followed.

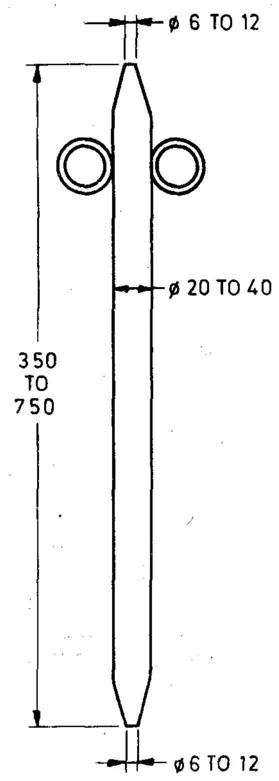

FIG. 2 SAMPLING TUBE

H-4 COMPOSITE SAMPLE

H-4.1 From Tanks and Drums

As far as possible, samples from a tank or drum should be drawn during the operation of filling. In that case equal amounts of the material shall be collected at regular intervals so as to get a total amount of about 1 500 ml. Where it is not possible to take a sample during filling, the material shall be drawn from different positions and depths with the sampling bottle or can after thoroughly agitating the material so as to ensure a fair amount of homogenity. The total amount of material collected shall be thoroughly mixed and divided into three equal portions, one for the purchaser, another for the manufacturer and the third for the referee.

H-4.2 From Bottles and Small Containers

From each of the bottle or container selected according to **H-3.2.3** a small representative portion of the material shall be drawn with the help of the sampling tube. Equal quantities of the material so drawn from the various containers shall be thoroughly mixed to form a test sample of about 1 500 ml. This shall be divided into three equal parts, one for the purchaser, another for the manufacturer and the third for the referee.

H-4.3 All the tests samples shall be transferred to separate sample containers and sealed and labelled with full identification particulars. The referee test sample, bearing the seal of both the purchaser and the manufacturer shall be kept at a place agreed to between the two and shall be used in case of dispute.

H-5 TESTS

H-5.1 Tests for the determination of all the requirements given in this specification shall be performed on the test sample as obtained in **H-4.1** or **H-4.2**

H-6 CRITERIA FOR CONFORMITY

H-6.1 The lot shall be declared as conforming to this specification if all the test results satisfy the requirements prescribed in **2** and Table 1.

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